Indian J. Phys. **78A** (1), 89-91 (2004)

Nanomaterials–2003

AN INTERNATIONAL JOURNAL IJP

Magnetic and current density studies on nanostructured vanadium nitride material

Raghumani Singh Ningthoujam and N S Gajbhiye*

Department of Chemistry, Indian Institute of Technology, Kanpur-208 016, Uttar Pradesh, India

E-mail : nsg@iitk.ac.in

Abstract : Nanostructured vanadium nitride material is prepared by chemical synthesis route. Atomic force microscopy study shows the particle size of 40 nm. Topography of the surface is studied. This shows the superconducting property at T_c ~9.2 K which can be confirmed from the electrical resistivity studies. The magnetic data is fitted by using $\chi = \chi_0 + C/T$ in temperature range 10–300 K indicating Curie-law at low temperatures and Pauli-paramagnetism at high temperatures. Critical current density value is found to be 68 A/cm² which is the minimum limit of current density that can destroy the superconducting property

Keywords : Nanostructured vanadium nitride material, superconducting materials, critical current density, magnetic susceptibility, nanocrystallinity.

PACS Nos. : 74.70.-b, 75.75.+a, 81.07.Bc

1. Introduction

Most transition metal nitrides and carbides are the metallic, catalytic, hard, coating, magnetic and superconducting materials [1,2]. The critical current density measurements on superconducting materials are very important for superconducting magnetic applications [3]. The critical current density measurements on nitrides – NbN, ZrN, TiN, Nb-Zr-N, Nb-Ti-N, and carbides – NbC, TaC, Mo_2C, Mo_3Al_2C on the thin film and bulk are reported [1,4]. The critical current density depends on thickness, nonmetal-to-metal ratio, microstructure and the method of preparation in nitrides and carbides.

Here, magnetic and critical current density studies of the nanostructured vanadium nitride (VN) are presented, and its values are compared with the thin film and bulk polycrystalline materials. Also, the surface topography study is presented here by using Atomic Force Microscopy (AFM).

2. Experimental

Earlier we prepared the nanostructured vanadium nitride material by the simultaneous decomposition and nitridation of $[VO(NH_2O)_2Gly].H_2O$ complex at 973 K for 4 h in NH_3 gas atmosphere [5]. Now, this material is prepared at much lower temperature ~723 K by using the bigger single crystalline of the same precursor and the slow heating rate. Here, heating rate is 2°C/min.

The characterization of this material is done by using the X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM), chemical analysis, the electrical four point probe method, and SQUID techniques. The magnetic measurement is done using a cylindrical shape of sample with diameter 0.3 cm and height 0.4 cm by using SQUID technique. Here, the rectangle shape of VN sample having dimensions of 1 cm length, 0.172 cm breadth and 0.072 cm thickness is for the electrical resistivity measurement by using the standard four point probe method. It should be taken care for making the rectangular shape of sample. The length of the sample should be much larger than breadth and thickness of sample. As possible as it should be thin.

3. Results and discussion

3.1. XRD study :

The XRD pattern of VN obtained at 723 K is shown in Figure 1; and it shows the cubic NaCl structure with lattice constant a = 4.138 Å. The crystallite sizes of 8–32 nm are obtained in the range of the preparation temperature 723–973 K, and it gives the nanocrystallinity of VN material.

* Corresponding Author

3.2. AFM study :

Two and three dimensional topography images of VN obtained at 973 K using AFM are shown in Figure 2(a) and (b) respectively. The shape of the particles is spherical with average particle size of 40 nm which is almost same with our previous reported values of TEM (35 nm) and XRD (32 nm) particle size [5]. The whole area of the surface is 360000 nm^2. The vertical surface can be characterized by the root mean square (rms) surface roughness (σ), and σ is found to be 10.19 nm.

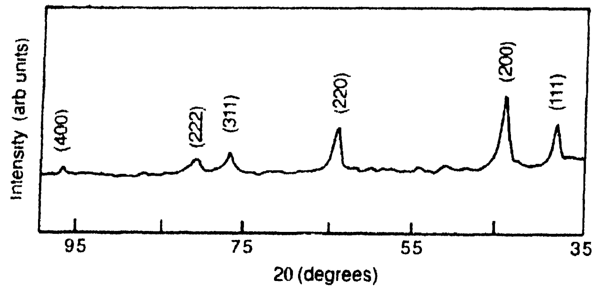

Figure 1. XRD pattern of VN which is prepared at 723 K. It shows the cubic structure

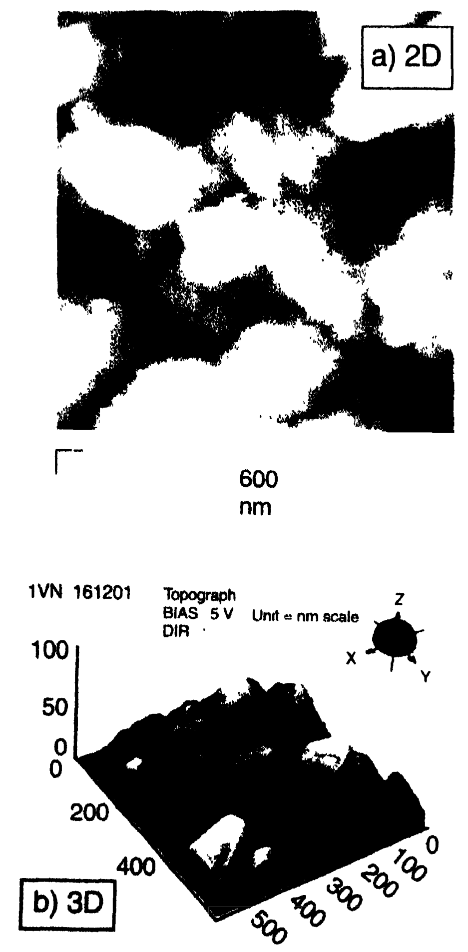

Figure 2. AFM surface topography images of VN sample. The 3- and 2-dimensional structures are shown as (a) and (b) respectively. The particle size is about 40 nm.

3.3 Magnetic study :

Figure 3 shows the magnetic susceptibility (χ) *versus* temperature (T) for 4–300 K. It gives the superconducting to normal transition (T_c) at ~8.6 K. Above T_c (say 10–300 K), there is a constancy of χ with the temperature. However, the careful study shows that χ is dependent on temperature. The susceptibility (χ) data are fitted in the following equation :

$$\chi = \chi_0 + C/T .$$

The value of χ_0 is found to be 2.18 × 10^{-6} emu/g/Oe, and the Curie constant C ~2 × 10^{-5} emu/g/Oe/K^{-1}. The fitted curve to the data points is shown in Figure 3 as inset. It indicates that the Curie-law is followed at low temperatures, and Pauli paramagnetism at high temperatures.

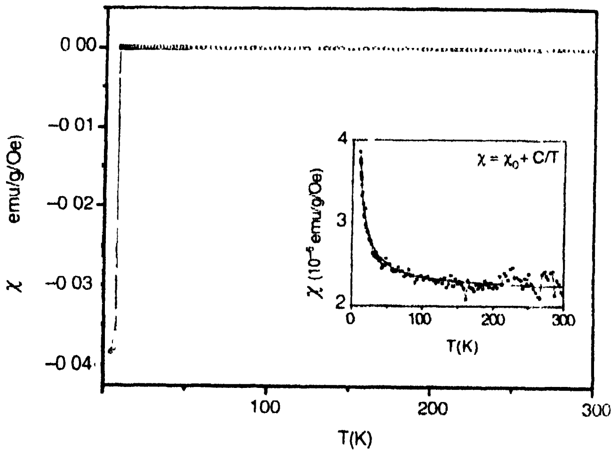

Figure 3. Magnetic susceptibility (χ) *vs* Temperature (T) of VN sample to show superconducting transition. The inset shows the fitted curve to magnetic data points at above T_c, *i.e* in between 10–300 K

The room temperature (χ_{300}) value in our VN sample is 138 × 10^{-6} emu/mole/Oe, but the thin film and polycrystalline VN reported values are about 240 × 10^{-6} emu/mole/Oe [1,6] where paramagnetic spin fluctuations arising from the high susceptibility (χ) or high electron density (D_f) at Fermi-level reduce the theoretical T_c ~30 K to the experimental value ~8.6 K The D_f is directly proportional to χ . D_f is found to be 3.81 × 10^{34} cm^{-3}erg^{-1}. Also, this reduction of T_c gives the low temperature resistivity study to the T^2- power law [6]. Here, T^2- and T^3-laws are due to electron-electron (*e-e*) and electron-phonon (*e-ph*) interactions respectively. However, our VN sample has the less susceptibility value or less electron density states at the fermi-level than the reported value. Due to this the *e-ph* interactions are predominant compared to *e-e* interactions. Such results are found in our previous studies of electrical resistivity [5]. Low temperature electrical resistivity studies on this VN found that it follows the T^3-power law indicating *e-ph* interactions predominantly in the temperature range 10–30 K [5].

3.4. Critical current density study :

Our previous electrical resistivity studies on nanocrystalline VN show T_c at ~9.2 K [5] which is closed to the magnetic value

(8.6 K). Our T_c ~9.2 K is slightly greater than the reported values of thin and polycrystalline VN materials (8–9 K) [6]. It may be arose from the surface smoothness which can be found from AFM study. For the critical current density measurement, the minimum five different currents are passed through the sample. The superconducting to normal transition (T_c) is measured from resistivity *vs* temperature curve. The different T_c values with currents are shown in Figure 4. The currents (I) are converted to the current densities (J). The current density is defined as the

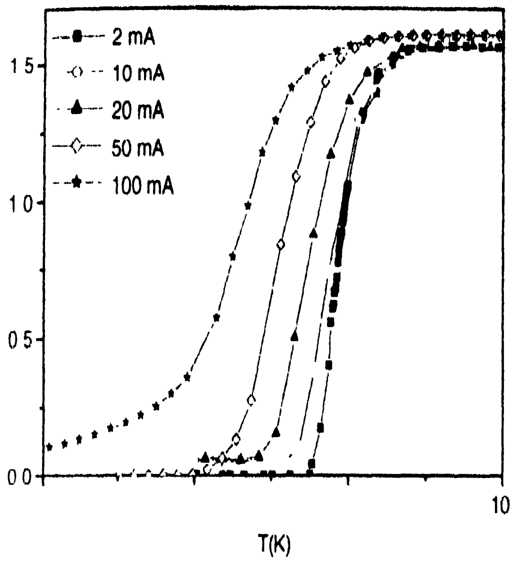

Figure 4. The resistivity (ρ) *vs* Temperature (T) below T_c (say 10 K) for the different currents for VN sample. Here, minimum 5 different currents taken. T_c shifts to lower value with increase of currents

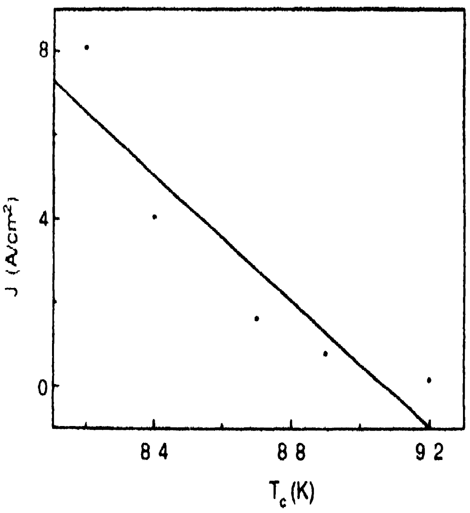

Figure 5. Current density (J) *vs* T_c for VN sample. The experimental data points are shown here, and linear line is fitted to data points

amount of the electrical currents flowing through a solid per unit area (measured in the direction perpendicular to the flow direction). We plot the current density (J) *vs* T_c as shown in Figure 5. A linear fit to the data points is done. The critical current density (J_c) which is taken by an appropriate extrapolation (linear fit) to T_c = 0 K is 68 A/cm^2. This value is less than the reported values of other thin film nitrides [1]. Such type of decrease of J_c for bulk compared to those of thin film or wire was reported [1].

4. Conclusion

AFM study on the nanostructured VN which is prepared at 973 K shows the spherical shape of 40 nm particle size. It shows the superconducting property. Magnetic data is fitted well using $\chi = \chi_0 + C/T$. The standard four point probe should be used for the determination of absolute resistivity and critical current density. The critical current density (J_c) is of 68 mA/cm^2.

Acknowledgment

DST, New Delhi and Chemistry Department, IIT Kanpur are highly acknowledged for the financial support during the investigation. Also, RSN thank N Sudhakar of IIT Kanpur, and Dr. A K Nigam of TIFR, Mumbai for help to get the resistivity and magnetic data respectively; and Sanjay, IIT Kanpur for technical correction.

References

[1] L E Toth *Transition Metal Carbides and Nitrides* (New York : Academic) (1971)

[2] N S Gajbhiye, R S Ningthoujam and J Weissmuller *Phys. Stat. Sol.* **A189** 691 (2002)

[3] R T Kampwirth, D W Capone II, K E Gray and A Vicens *IEEE Trans on Mag* **MAG-21** 459 (1985)

[4] J R Gavaler, M A Janocko, A Patterson and C K Jones *J Appl. Phys.* **42** 54 (1971)

[5] R S Ningthoujam, N Sudhakar, K P Rajeev and N S Gajbhiye *J. Appl Phys.* **91** 6051 (2002)

[6] H Rietschel, H Winter and W Reichardt *Phys. Rev* **B22** 4284 (1980)